EXHIBIT 2

POM=0.003355×VOCE

POM=Polycyclic organic matter emissions in terms of milligrams per mile

VOCE=Non-methane, non-ethane exhaust emissions of volatile organic compounds in grams per mile.

(9) Nonexhaust benzene emissions in VOC Control Region 1 shall be given by the following equations for both Phase I and Phase II:

NEBZ1=DIBZ1+HSBZ1+RLBZ1+RFBZ1

HSBZ1=10 × BEN × HSVOC1 × [(−0.0342 × MTB)+(−0.080274 × RVP)+1.4448]

DIBZ1=10 × BEN × DIVOC1 × [(−0.0290 × MTB)+(−0.080274 × RVP)+1.3758]

RLBZ1=10 × BEN × RLVOC1 × [(−0.0342 × MTB)+(−0.080274 × RVP)+1.4448]

RFBZ1=10 × BEN × RFVOC1 × [(−0.0296 × MTB)+(−0.081507 × RVP)+1.3972]

where

NEBZ1=Nonexhaust emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile.

DIBZ1=Diurnal emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile.

HSBZ1=Hot soak emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile.

RLBZ1=Running loss emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile.

RFBZ1=Refueling emissions of volatile organic compounds in VOC Control Region 1 in grams per mile.

VOCDI1=Diurnal emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile, as determined in paragraph (c)(3) of this section.

VOCHS1=Hot soak emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile, as determined in paragraph (c)(3) of this section.

VOCRL1=Running loss emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile, as determined in paragraph (c)(3) of this section.

VOCRF1=Refueling emissions of volatile organic compounds in VOC Control Region 1 in milligrams per mile, as determined in paragraph (c)(3) of this section.

(10) Nonexhaust benzene emissions in VOC Control Region 2 shall be given by the following equations for both Phase I and Phase II:

NEBZ2=DIBZ2+HSBZ2+RLBZ2+RFBZ2

HSBZ2=10 ×BEN × HSVOC2 × [(−0.0342 × MTB)+(−0.080274 × RVP)+1.4448]

DIBZ2=10 × BEN × DIVOC2 × [(−0.0290 × MTB)+(−0.080274 × RVP)+1.3758]

RLBZ2=10 × BEN × RLVOC2 × [(−0.0342 × MTB)+(−0.080274 × RVP)+1.4448]

RFBZ2=10 × BEN × RFVOC2 × [(−0.0296 × MTB)+(−0.081507 × RVP)+1.3972]

where

NEBZ2=Nonexhaust emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile.

DIBZ2=Diurnal emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile.

HSBZ2=Hot soak emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile.

RLBZ2=Running loss emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile.

RFBZ2=Refueling emissions of volatile organic compounds in VOC Control Region 2 in grams per mile.

VOCDI2=Diurnal emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile, as determined in paragraph (c)(4) of this section.

VOCHS2=Hot soak emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile, as determined in paragraph (c)(4) of this section.

VOCRL2=Running loss emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile, as determined in paragraph (c)(4) of this section.

VOCRF2=Refueling emissions of volatile organic compounds in VOC Control Region 2 in milligrams per mile, as determined in paragraph (c)(4) of this section.

(f) *Limits of the model.* (1) The equations described in paragraphs (a), (c), and (d) of this section shall be valid only for fuels with fuel properties that fall in the following ranges for reformulated gasolines and conventional gasolines:

(i) For reformulated gasolines:

Fuel property	Acceptable range
Oxygen	0.00–3.70 weight percent.
Sulfur	0.0–500.0 parts per million by weight.
RVP	6.4–10.0 pounds per square inch.
E200	30.0–70.0 volume percent.
E300	70.0–100.0 volume percent.
Aromatics	0.0–50.0 volume percent.
Olefins	0.00–25.0 volume percent.
Benzene	0.0–2.0 volume percent.

(ii) For conventional gasolines:

Fuel property	Acceptable range
Oxygen	0.00–3.70 weight percent.
Sulfur	0.0–1000.0 parts per million by weight.
RVP	6.4–11.0 pounds per square inch.
E200	30.0–70.0 volume percent.
E300	70.0–100.0 volume percent.
Aromatics	00.0–55.0 volume percent.
Olefins	0.00–30.0 volume percent.
Benzene	0.0–4.9 volume percent.

(2) Fuels with one or more properties that do not fall within the ranges described in above shall not be certified or evaluated for their emissions performance using the complex emissions model described in paragraphs (c), (d), and (e) of this section.

§ 80.46 Measurement of reformulated gasoline fuel parameters.

(a) *Sulfur.* Sulfur content shall be determined using American Society for Testing and Materials (ASTM) standard method D–2622–92, entitled "Standard Test Method for Sulfur in Petroleum Products by X-Ray Spectrometry."

(b) *Olefins.* Olefin content shall be determined using ASTM standard method D–1319–93, entitled "Standard Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption."

(c) *Reid vapor pressure (RVP).* Reid Vapor Pressure (RVP) shall be determined using the procedure described in 40 CFR part 80, appendix E, Method 3.

(d) *Distillation.* (1) Distillation parameters shall be determined using ASTM standard method D–86–90, entitled "Standard Test Method for Distillation of Petroleum Products"; except that

(2) The figures for repeatability and reproducibility given in degrees Fahrenheit in Table 9 in the ASTM method are incorrect, and shall not be used.

(e) *Benzene.* (1) Benzene content shall be determined using ASTM standard method D–3606–92, entitled "Standard Test Method for Determination of Benzene and Toluene in Finished Motor and Aviation Gasoline by Gas Chromatography"; except that

(2) Instrument parameters must be adjusted to ensure complete resolution of the benzene, ethanol and methanol

peaks because ethanol and methanol may cause interference with ASTM standard method D–3606–92 when present.

(f) *Aromatics.* Aromatics content shall be determined by gas chromatography identifying and quantifying each aromatic compound as set forth in paragraph (f)(1) of this section.

(1) (i) *Detector.* The detector is an atomic mass spectrometer detector (MSD). The detector may be set for either selective ion or scan mode.

(ii) *Method A.* (A) The initial study of this method used a three component internal standard using the following calculations.

(B) The calibration points are constructed by calculating an amount ratio and response ratio for each level of a particular peak in the instrument's calibration table.

(C) The amount ratio is the amount of the compound divided by the amount of the internal standard for a given level.

(D) The response ratio is the response of the compound divided by the response of the internal standard at this level.

(E) The equation for the curve through the calibration points is calculated using the type fit and origin handling specified in the instrument's calibration table. In the initial study the fit was a

second degree polynomial including a forced zero for the origin.

(F) The response of the compound in a sample is divided by the response of the internal standard to provide a response ratio for that compound in the sample.

(G) A corrected amount ratio for the unknown is calculated using the curve fit equation determined in paragragh (f)(1)(ii)(E) of this section.

(H) The amount of the aromatic compound is equal to the corrected amount ratio times the Amount of Internal Standard.

(I) The total aromatics in the sample is the sum of the amounts of the individual aromatic compounds in the sample.

(J) An internal standard solution can be made with the following compounds at the listed concentrations in volume percent. Also listed is the Chemical Abstracts Service Registry Number (CAS), atomic mass unit (amu) on which the detector must be set at the corresponding retention time if used in the selective ion mode, retention times in minutes, and boiling point in °C. (Other, similar, boiling point materials can be used which are not found in gasoline.) Retention times are approximate and apply only to a 60 meter capillary column used in the

initial study. Other columns and retention times can be used.

(1) 4-methyl-2-pentanone, 50 vol% [108–10–1], 43.0 amu, 22.8 min., bp 118;

(2) benzyl alcohol, 25 vol%, [100–51–6], 108 amu, 61.7 min., bp 205;

(3) 1-octanol, [111–87–5], 25 vol%, 56.0 amu, 76.6 min., bp 196;

(K) At least two calibration mixtures which bracket the measured total aromatics concentration must be made with a representative mixture of aromatic compounds. The materials and concentrations used in the highest concentration calibration level in the initial study for this method are listed in this paragraph (f)(1)(ii)(K). Also listed is the Chemical Abstracts Service Registry Number (CAS), atomic mass unit (amu) on which the detector must be set for the corresponding retention time if used in the selective ion mode, retention times in minutes, and in some cases boiling point in °C. The standards are made in 2,2,4-trimethylpentane (iso-octane), [540–84–1]. Other aromatic compounds, and retention times may be acceptable as long as the aromatic values produced meet the criteria found in the quality assurance section for the aromatic methods.

Compound	Con-centra-tions vol %	CAS No.	AMU	Retention time, min.	Boiling point, °C
Benzene	2.25	71–43–2	78	18.9	80.1
Methylbenzene	2.5	108–88–3	91	25.5	111
Ethylbenzene	2.25	100–41–4	91	34.1	136.2
1,3–Dimethylbenzene 1,4–Dimethylbenzene	5	108–38–3	91	35.1	136–138
1,2-dimethylbenzene	10	95–47–6	91	38.1	144
(1-methylethyl)-benzene	2.25	620–14–4	105	42.8	....
Propylbenzene	2.25	103–65–1	91	48.0	159.2
1-ethyl-2-methylbenzene	2.25	611–14–3	105	49.3	165
1,2,4-trimethylbenzene	2.25	95–63–6	105	50.9	169
1-ethyl-2-methylbenzene	2.25	611–14–4	105	53.3	165
1,3-diethylbenzene	2.25	141–93–5	119	56.6	181
Butylbenzene	2.25	104–51–8	91	60.7	183
1-methyl-2-(1-methylethyl)-benzene	2.25	933–98–2	119	63.9	....
1-ethyl-3-methylbenzene	2.25	620–14–4	105	64.2	....
1-methyl-4-iso-propylbenzene	2.25	99–87–6	119	69.0	177
2-ethyl-1,3-dimethylbenzene	2.25	2870–04–4	119	73.0	....
2-methylpropylbenzene	2.25	538–93–2	91	75.0	....
1-methyl-3-(1-methylethyl)-benzene	2.25	535–77–3	119	75.6	....
1-methyl-3-propylbenzene	2.25	1074–43–7	105	78.9	....
2-ethyl-1,4-dimethylbenzene	2.25	1758–88–9	119	83.2	187
1-methyl-4-(methylethyl)-benzene	2.25	934–80–9	119	83.4	....
1-ethyl-2,4-dimethylbenzene	2.25	874–41–9	119	85.7	....
(1,1-dimethylethy)-3-methylbenzene	2.25	27138-21–2	133	87.3	....
1-ethyl-2,3-dimethylbenzene	2.25	933–98–2	119	88.7	....
1-(1,1-dimethylethyl)-3-methylbenzene	2.25	175–38–3	133	89.4	....
1-ethyl-1,4-dimethylbenzene	2.25	874–41–9	119	94.9	....
2-ethyl-1,3-dimethylbenzene	2.25	2870–04–4	119	100.9	....
1-ethyl-3,5-dimethylbenzene	2.25	934–74–7	119	102.5	....
1–2,4,5-tetramethylbenzene	2.25	95–93–2	119	115.9	197
Pentylbenzene	2.25	538–68–1	91	116	....
Naphthalene	2.25	191–20–3	128	118.4	198
3,5 dimethyl-iso-butylbenzene	2.25	98–19–1	147	118.5	205.5

(iii) *Method B*. (A) Use a percent normalized format to determine the concentration of the individual compounds. No internal standard is used in this method.

(B) The calculation of the aromatic compounds is done by developing calibration curves for each compound using the type fit and origin handling specified in the instrument's calibration table.

(C) The amount of compound in a sample (the corrected amount) is calculated using the equation determined in paragraph (f)(1)(ii) of this section for that compound.

(D) The percent normalized amount of a compound is calculated using the following equation:

$$A_n = 100 \times \left(\frac{A_c}{A_s} \right)$$

where:

A_n = percent normalized amount of a compound
A_c = corrected amount of the compound
A_s = sum of all the corrected amounts for all identified compounds in the sample

(E) The total aromatics is the sum of all the percent normalized aromatic amounts in the sample.

(F) This method allows quantification of non-aromatic compounds in the sample. However, correct quantification can only be achieved if the instrument's calibration table can identify the compounds that are responsible for at least 95 volume percent of the sample and meets the following quality control criteria.

(2) *Quality assurance*. (i) The performance standards will be from repeated measurement of the calibration mixture, standard reference material, or process control gasoline. The uncertainty in the measured aromatics percentages in the standards must be less than 2.0 volume percent in the fuel at a 95% confidence level.

(ii) If the bias of the standard mean is greater than 2% of the theoretical value, then the standard measurement and measurements of all samples measured subsequent to the previous standard measurement that met the performance criteria must be repeated after re-calibrating the instrument.

(iii) Replicate samples must be within 3.0 volume percent of the previous sample or within 2.0 volume percent of the mean at the 95% confidence level.

(3) *Alternative test method*. (i) Prior to January 1, 1997, any refiner or importer may determine aromatics content using ASTM standard method D–1319–93, entitled "Standard Test Method for Hydrocarbon Types in Liquid Petroleum Products by Fluorescent Indicator Adsorption," for purposes of meeting any testing requirement involving aromatics content; provided that

(ii) The refiner or importer test result is correlated with the method specified in paragraph (f)(1) of this section.

(g) *Oxygen and oxygenate content analysis*. Oxygen and oxygenate content shall be determined by the gas chromatographic procedure using an oxygenate flame ionization detector (GC-OFID) as set out in paragraphs (g) (1) through (8) of this section.

(1) *Introduction; scope of application*. (i) The following single-column, direct-injection gas chromatographic procedure is a technique for quantifying the oxygenate content of gasoline.

(ii) This method covers the quantitative determination of the oxygenate content of gasoline through the use of an oxygenate flame ionization detector (OFID). It is applicable to individual organic oxygenated compounds (up to 20 mass percent each) in gasoline having a final boiling point not greater than 220 °C. Samples above this level should be diluted to fall within the specified range.

(iii) The total concentration of oxygen in the gasoline, due to oxygenated components, may also be determined with this method by summation of all peak areas except for dissolved oxygen, water, and the internal standard. Sensitivities to each component oxygenate must be incorporated in the calculation.

(iv) All oxygenated gasoline components (alcohols, ethers, etc.) may be assessed by this method.

(v) The total mass percent of oxygen in the gasoline due to oxygenated components also may be determined with this method by summing all peak areas except for dissolved oxygen, water, and the internal standard.

(vi) Where trade names or specific products are noted in the method, equivalent apparatus and chemical reagents may be used. Mention of trade names or specific products is for the assistance of the user and does not constitute endorsement by the U.S. Environmental Protection Agency.

(2) *Summary of method*. A sample of gasoline is spiked to introduce an internal standard, mixed, and injected into a gas chromatograph (GC) equipped with an OFID. After chromatographic resolution the sample components enter a cracker reactor in which they are stoichiometrically converted to carbon monoxide (in the case of oxygenates), elemental carbon, and hydrogen. The carbon monoxide then enters a methanizer reactor for conversion to water and methane. Finally, the methane generated is determined by a flame ionization detector (FID).

(3) *Sample handling and preservation*. (i) Samples shall be collected and stored in containers which will protect them from changes in the oxygenated component contents of the gasoline, such as loss of volatile fractions of the gasoline by evaporation.

(ii) If samples have been refrigerated they shall be brought to room temperature prior to analysis.

(iii) Gasoline is extremely flammable and should be handled cautiously and with adequate ventilation. The vapors are harmful if inhaled and prolonged breathing of vapors should be avoided. Skin contact should be minimized.

(4) *Apparatus*. (i) A GC equipped with an oxygenate flame ionization detector.

(ii) An autosampler for the GC is highly recommended.

(iii) A 60-m length, 0.25-mm ID, 1.0-μm film thickness, nonpolar capillary GC column (J&W DB–1 or equivalent) is recommended.

(iv) An integrator or other acceptable system to collect and process the GC signal.

(v) A positive displacement pipet (200 μL) for adding the internal standard.

(5) *Reagents and materials*. Gasoline and many of the oxygenate additives are extremely flammable and may be toxic over prolonged exposure. Methanol is particularly hazardous. Persons performing this procedure must be familiar with the chemicals involved and all precautions applicable to each.

(i) Reagent grade oxygenates for internal standards and for preparation of standard solutions.

(ii) Supply of oxygenate-free gasoline for blank assessments and for preparation of standard solutions.

(iii) Calibration standard solutions containing known quantities of suspected oxygenates in gasoline.

(iv) Calibration check standard solutions prepared in the same manner as the calibration standards.

(v) Reference standard solutions containing known quantities of suspected oxygenates in gasoline.

(vi) Glass standard and test sample containers (between 5 and 100 Ml capacity) fitted with a self-sealing polytetrafluoroethlene (PTFE) faced rubber septum crimp-on or screw-down sealing cap for preparation of standards and samples.

(6) *Calibration*.—(i)(A) Calibration standards of reagent-grade or better oxygenates (such as methanol, absolute ethanol, methyl t-butyl ether (MTBE), di-i-propyl ether (DIPE), ethyl t-butyl ether (ETBE), and t-amyl methyl ether (TAME)) are to be prepared

gravimetrically by blending with gasoline that has been previously determined by GC/OFID to be free of oxygenates. Newly acquired stocks of reagent grade oxygenates shall be analyzed for contamination by GC/FID and GC/OFID before use.

(B) Required calibration standards (percent by volume in gasoline):

Oxygenate	Range (percent)	Number of stand-ards (min-imum)
Methanol	0.25–12.00	5
Ethanol	0.25–12.00	5
t-Butanol	0.25–12.00	5
MTBE	0.25–15.00	5

(ii) Take a glass sample container and its PTFE faced rubber septum sealing cap. Transfer a quantity of an oxygenate to the sample container and record the mass of the oxygenate to the nearest 0.1 mg. Repeat this process for any additional oxygenates of interest except the internal standard. Add oxygenate-free gasoline to dilute the oxygenates to the desired concentration. Record the mass of gasoline added to the nearest 0.1 mg, and determine and label the standard according to the mass percent quantities of each oxygenate added. These standards are not to exceed 20 mass percent for any individual pure component due to potential hydrocarbon breakthrough and/or loss of calibration linearity.

(iii) Inject a quantity of an internal standard (such as 2-butanol) and weigh the contents again. Record the difference in masses as the mass of internal standard to the nearest 0.1 mg. The mass of the internal standard shall amount to between 2 and 6 percent of the mass of the test sample (standard). The addition of an internal standard reduces errors caused by variations in injection volumes.

(iv) Ensure that the prepared standard is thoroughly mixed and transfer approximately 2 Ml of the solution to a vial compatible with the autosampler if such equipment is used.

(v) At least five concentrations of each of the expected oxygenates should be prepared. The standards should be as equally spaced as possible within the range and may contain more than one oxygenate. A blank for zero concentration assessments is also to be included. Additional standards should be prepared for other oxygenates of concern.

(vi) Based on the recommended chromatographic operating conditions specified in paragraph (g)(7)(i) of this section, determine the retention time of each oxygenate component by analyzing

dilute aliquots either separately or in known mixtures. Reference should be made to the Chemical Abstracts Service (CAS) registry number of each of the analytes for proper identification. Approximate retention times for selected oxygenates under these conditions are as follows:

Oxygenate	CAS	Reten-tion time (min-utes)
Dissolved oxygen .	7782–44–7	5.50
Water	7732–18–5	7.20
Methanol	67–56–1	9.10
Ethanol	64–17–5	12.60
Propanone	67–64–1	15.00
2–Propanol	67–63–0	15.70
t-Butanol	75–65–0	18.00
n-Propanol	71–23–8	21.10
MTBE	1634–04–4	23.80
2–Butanol	15892–23–6	26.30
i-Butanol	78–83–1	30.30
ETBE	637–92–3	31.10
n-Butanol	71–36–3	33.50
TAME	994–05–8	35.30
i-Pentanol	137–32–6	38.10

(vii) By GC/OFID analysis, determine the peak area of each oxygenate and of the internal standard.

(viii) Obtain a calibration curve by performing a least-squares fit of the relative area response factors of the oxygenate standards to their relative mass response factors as follows:

$R_{ao}=b_oR_{mo}+b_1(R_{mo})^2$

where:

R_{ao} = relative area response factor of the oxygenate, A_o/A_i

R_{mo} = relative mass response factor of the oxygenate, M_o/M_i

A_o = area of the oxygenate peak

A_i = area of the internal standard peak

M_o = mass of the oxygenate added to the calibration standard

M_i = mass of internal standard added to the calibration standard

b_0 = linear regression coefficient

b_1 = quadratic regression coefficient

(7) *Procedure.* (i) GC operating conditions:

(A) Oxygenate-free helium carrier gas: 1.1 Ml/min (2 bar), 22.7 cm/sec at 115 °C;

(B) Carrier gas split ratio: 1:100;

(C) Zero air FID fuel: 370 Ml/min (2 bar);

(D) Oxygenate free hydrogen FID fuel: 15 Ml/min (2 bar);

(E) Injector temperature: 250 °C;

(F) Injection volume: 0.5 μL;

(G) Cracker reactor temperature: sufficiently high enough temperature to ensure reduction of all hydrocarbons to the elemental states (i.e., $C_xH_{2x} \rightarrow C + H_2$, etc.);

(H) FID temperature: 400 °C; and

(I) Oven temperature program: 40 °C for 6 min, followed by a temperature increase of 5 °C/min to 50 °C, hold at 50 °C for 5 min, followed by a temperature increase of 25 °C/min to 175 °C, and hold at 175 °C for 2 min.

(ii) Prior to analysis of any samples, inject a sample of oxygenate-free gasoline into the GC to test for hydrocarbon breakthrough overloading the cracker reactor. If breakthrough occurs, the OFID is not operating effectively and must be corrected before samples can be analyzed.

(iii) Prepare gasoline test samples for analysis as follows:

(A) Tare a glass sample container and its PTFE faced rubber septum sealing cap. Transfer a quantity of the gasoline sample to the sample container and record the mass of the transferred sample to the nearest 0.1 mg.

(B) Inject a quantity of the same internal standard (such as 2-butanol) used in generating the standards and weigh the contents again. Record the difference in masses as the mass of internal standard to the nearest 0.1 mg. The mass of the internal standard shall amount to between 2 and 6 percent of the mass of the test sample (standard). The addition of an internal standard reduces errors caused by variations in injection volumes.

(C) Ensure that this test sample (gasoline plus internal standard) is thoroughly mixed and transfer approximately 2 mL of the solution to a vial compatible with the autosampler if such equipment is used.

(iv) After GC/OFID analysis, identify the oxygenates in the sample based on retention times, determine the peak area of each oxygenate and of the internal standard, and calculate the relative area response factor for each oxygenate.

(v) Monitor the peak area of the internal standard. A larger than expected peak area for the internal standard when analyzing a test sample may indicate that this oxygenate is present in the original sample. Prepare a new aliquot of the sample without addition of the oxygenate internal standard. If the presence of the oxygenate previously used as the internal standard can be detected, then either:

(A) The concentration of this oxygenate must be assessed by the method of standard additions; or

(B) An alternative internal standard, based on an oxygenate that is not present in the original sample, must be utilized with new calibration curves.

(vi) Calculate the relative mass response factor (R_{mo}) for each oxygenate based on the relative area response

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factor (R_{ao}) and the calibration equation in paragraph (g)(6)(viii) of this section.

(vii) Calculate the mass percent of the oxygenate in the test sample according to the following equation:

$$M_o\% = \frac{R_{mo}M_i(100\%)}{M_s}$$

where:

$M_o\%$ = mass percent of the oxygenate in the test sample

M_s = mass of sample to which internal standard is added

(viii) If the mass percent exceeds the calibrated range, gravimetrically dilute a portion of the original sample to a concentration within the calibration range and analyze this sample starting with paragraph (g)(7)(iii) of this section.

(ix) Report the total weight percent oxygen as follows:

(A) Subtract the peak areas due to dissolved oxygen, water, and the internal standard from the total summed peak areas of the chromatogram.

(B) Assume the total summed peak area solely due to one of the oxygenates that the instrument is calibrated for and determine the total mass percent as that oxygenate based on paragraph (g)(7)(vii) of this section. For simplicity, chose an oxygenate having one oxygen atom per molecule.

(C) Multiply this concentration by the molar mass of oxygen and divide by the molar mass of the chosen oxygenate to determine the mass percent oxygen in the sample. For example, if the total peak area is based on MTBE, multiply by 16.00 (the molar mass of atomic oxygen) and divide by 88.15 (the molar mass of MTBE).

(x) Sufficient sample should be retained to permit reanalysis.

(8) *Quality control procedures and accuracy.* (i) The laboratory shall routinely monitor the repeatability (precision) of its analyses. The recommendations are:

(A) The preparation and analysis of laboratory duplicates at a rate of one per analysis batch or at least one per ten samples, whichever is more frequent.

(B) Laboratory duplicates shall be carried through all sample preparation steps independently.

(C) The range (R) for duplicate samples should be less than the following limits:

Oxygenate	Concentration mass percent	Upper limit for range mass percent
Methanol	0.27–1.07	0.010+0.043C
Methanol	1.07–12.73	0.053C
Ethanol	1.01–12.70	0.053C
MTBE	0.25–15.00	0.069+0.029C
DIPE	0.98–17.70	0.048C
ETBE	1.00–18.04	0.074C
TAME	1.04–18.59	0.060C

where:

$C=(C_o+C_d)/2$

C_o=concentration of the original sample

C_d=concentration of the duplicate sample

R=Range, $|C_o-C_d|$

(D) If the limits in paragraph (g)(8)(i)(C) of this section are exceeded, the sources of error in the analysis should be determined, corrected, and all analyses subsequent to and including the last duplicate analysis confirmed to be within the compliance specifications must be repeated. The specification limits for the range and relative range of duplicate analyses are minimum performance requirements. The performance of individual laboratories may indeed be better than these minimum requirements. For this reason it is recommended that control charts be utilized to monitor the variability of measurements in order to optimally detect abnormal situations and ensure a stable measurement process.

(E) *(1)* For reference purposes, a single laboratory study of repeatability was conducted on approximately 27

replicates at each of five concentrations for each oxygenate. The variation of MTBE analyses as measured by standard deviation was very linear with respect to concentration. Where concentration is expressed as mass percent, over the concentration range of 0.25 to 15.0 mass percent this relationship is described by the equation:

standard deviation=0.00784×C+0.0187

(2) The other oxygenates of interest, methanol, ethanol, DIPE, ETBE, and TAME, had consistent coefficients of variation at one mass percent and above:

Oxygenate	Concentration mass percent	Coefficient of variation percent of point
Methanol	1.07–12.73	1.43
Ethanol	1.01–12.70	1.43
DIPE	0.98–17.70	1.29
ETBE	1.00–18.04	2.00
TAME	1.04–18.59	1.62

(3) The relationship of standard deviation and concentration for methanol between 0.27 and 1.07 mass percent was very linear and is described by the equation:

standard deviation=0.0118×C+0.0027

(4) Based on these relationships, repeatability for the selected oxygenates at 2.0 and 2.7 mass percent oxygen were determined to be as follows, where repeatability is defined as the half width of the 95 percent confidence interval (i.e., 1.96 standard deviations) for a single analysis at the stated concentration:

Oxygenate	Concentration			Repeatability mass percent
	Mass percent oxygen	Mass percent oxygenate	Volume percent oxygenate	
Methanol	2.0	4.00	3.75	0.11
Ethanol	2.0	5.75	5.41	0.16
MTBE	2.00	11.00	11.00	0.21
DIPE	2.0	12.77	13.00	0.32
ETBE	2.0	12.77	12.74	0.50

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Oxygenate	Concentration			Repeatability mass per- cent
	Mass percent oxygen	Mass percent oxy- genate	Volume percent oxy- genate	
TAME	2.0	12.77	12.33	0.41
Methanol	2.7	5.40	5.07	0.15
Ethanol	2.7	7.76	7.31	0.21
MTBE	2.7	14.88	14.88	0.26
DIPE	2.7	17.24	17.53	0.43
ETBE	2.7	17.24	17.20	0.67
TAME	2.7	17.24	16.68	0.55

(ii) The laboratory shall routinely monitor the accuracy of its analyses. The recommendations are:

(A) Calibration check standards and calibration standards may be prepared from the same oxygenate stocks and by the same analyst. However, calibration check standards and calibration standards must be prepared from separate batches of the final diluted standards. For the specification limits listed in paragraph (g)(8)(ii)(C) of this section, the concentration of the check standards should be in the range given in paragraph (g)(8)(i)(C) of this section.

(B) Calibration check standards shall be analyzed at a rate of at least one per analysis batch and at least one per 10 samples, whichever is more frequent.

(C) If the measured concentration of a calibration check standard is outside the range of 100.0% ±6.0% of the theoretical concentration for a selected oxygenate of 1.0 mass percent or above, the sources of error in the analysis should be determined, corrected, and all analyses subsequent to and including the last standard analysis confirmed to be within the compliance specifications must be repeated. The specification limits for the accuracy of calibration check standards analyses are minimum performance requirements. The

performance of individual laboratories may indeed be better than these minimum requirements. For this reason it is recommended that control charts be utilized to monitor the variability of measurements in order to optimally detect abnormal situations and ensure a stable measurement process.

(D) Independent reference standards should be purchased or prepared from materials that are independent of the calibration standards and calibration check standards, and must not be prepared by the same analyst. For the specification limits listed in paragraph (g)(8)(ii)(F) of this section, the concentration of the reference standards should be in the range given in paragraph (g)(8)(i)(C) of this section.

(E) Independent reference standards shall be analyzed at a rate of at least one per analysis batch and at least one per 100 samples, whichever is more frequent.

(F) If the measured concentration of an independent reference standard is outside the range of 100.0% ±10.0% of the theoretical concentration for a selected oxygenate of 1.0 mass percent or above, the sources of error in the analysis should be determined, corrected, and all analyses subsequent to and including the last independent

reference standard analysis confirmed to be within the compliance specifications in that batch must be repeated. The specification limits for the accuracy of independent reference standards analyses are minimum performance requirements. The performance of individual laboratories may be better than these minimum requirements. For this reason it is recommended that control charts be utilized to monitor the variability of measurements in order to optimally detect abnormal situations and ensure a stable measurement process.

(G) The preparation and analysis of spiked samples at a rate of one per analysis batch and at least one per ten samples.

(H) Spiked samples shall be prepared by adding a volume of a standard to a known volume of sample. To ensure adequate method detection limits, the volume of the standard added to the sample shall be limited to 5% or less than the volume of the sample. The spiked sample shall be carried through the same sample preparation steps as the background sample.

(I) The percent recovery of the spiked sample shall be calculated as follows:

$$\% \, Recovery = \frac{100\% \, (C_m(V_o + V_1) - C_o V_o)}{C_s \, V_1}$$

where:

V_o=Volume of sample (Ml)

V_1=Volume of spiking standard added (Ml)

C_m=Measured concentration of spiked sample

C_o=Measured background concentration of sample

C_s=Known concentration of spiking standard

(J) If the percent recovery of any individual spiked sample is outside the range 100% ±10% from the theoretical concentration, then the sources of error

in the analysis must be determined and corrected, and all analyses subsequent to and including the last analysis confirmed to be within the compliance specifications must be repeated. The maintenance of control charts is one acceptable method or ensuring compliance with this specification.

(K) *(1)* Either the range (absolute difference) or relative range (but not necessarily both) for duplicate samples shall be less than the following limits:

Oxygenate	Concentra- tion (volume percent)	Range	Rel- ative range (vol- ume per- cent)
Methanol	1.0–12.0	....	7.2
Ethanol	3.0–12.0	....	7.1
t-Butanol	3.0–12.0	....	9.4
MTBE	3.0–15.0	0.55	9.2

(2) Relative range is calculated as follows:

$$R_r = \frac{200(R)}{C_o + C_d}$$

where:

R_r=relative range
R=range
C_o=concentration of the original sample
C_d=concentration of the duplicate sample

(3) If the limits in paragraph (g)(8)(ii)(K)(*1*) of this section are exceeded, the sources of error in the analysis should be determined, corrected, and all analyses subsequent to and including the last duplicate analysis confirmed to be within the compliance specifications must be repeated. The specification limits for the range and relative range of duplicate analyses are minimum performance requirements. The performance of individual laboratories may indeed be better than these minimum requirements. For this reason it is recommended that control charts be utilized to monitor the variability of measurements in order to optimally detect abnormal situations and ensure a stable measurement process. For reference purposes, a single laboratory study of precision (approximately 35 replicates) yielded the following estimates of method precision:

Oxygenate	Con-centration (weight percent)	Repeatability (volume per-cent)	(Percent)
Methanol	2.0	3.7	0.11
Ethanol	2.0	5.4	0.24
t-Butanol	2.0	8.8	0.39
MTBE	2.0	11.0	0.37

(4) Repeatability is defined as the half width of the 95 percent confidence interval for a single analysis at the stated concentration.

(iii) The laboratory shall routinely monitor the accuracy of its analyses. At a minimum this shall include:

(A) Calibration check standards and calibration standards may be prepared from the same oxygenate stocks and by the same analyst. However, calibration check standards and calibration standards must be prepared from separate batches of the final diluted standards. For the specification limits listed in paragraph (g)(8)(iii)(C) of this section, the concentration of the check standards should be in the range given in paragraph (g)(8)(iii)(C) of this section.

(B) Calibration check standards shall be analyzed at a rate of one per analysis batch or at least one per ten samples, whichever is more frequent.

(C) If the measured concentration of a calibration check standard is outside the range of 100%±10% percent of the theoretical concentration for methanol and ethanol, or 100%±13% for t-butanol and MTBE, the sources of error in the analysis should be determined, corrected, and all analyses subsequent to and including the last standard analysis confirmed to be within the compliance specifications must be repeated. The specification limits for the accuracy of calibration check standards analyses are minimum performance requirements. The performance of individual laboratories may indeed be better than these minimum requirements. For this reason it is recommended that control charts be utilized to monitor the variability of measurements in order to optimally detect abnormal situations and ensure a stable measurement process.

(D) Independent reference standards shall be purchased or prepared from materials that are independent of the calibration standards and calibration check standards, and must not be prepared by the same analyst. For the specification limits listed in paragraph (g)(8)(iii)(F) of this section, the concentration of the reference standards should be in the range given in paragraph (g)(8)(iii)(C) of this section.

(E) Independent reference standards shall be analyzed at a rate of one per analysis batch or at least one per 100 samples, whichever is more frequent.

(F) If the measured concentration of an independent reference standard is outside the range of 100%±10% of the theoretical concentration for methanol and ethanol, or 100%±13% for t-butanol and MTBE, the sources of error in the analysis should be determined, corrected, and all analyses subsequent to and including the last independent reference standard analysis confirmed to be within the compliance specifications in that batch must be repeated. The specification limits for the accuracy of independent reference standards analyses are minimum performance requirements. The performance of individual laboratories may indeed be better than these minimum requirements. For this reason it is recommended that control charts be utilized to monitor the variability of measurements in order to optimally detect abnormal situations and ensure a stable measurement process.

(G) If matrix effects are suspected, then spiked samples shall be prepared and analyzed as follows:

(1) Spiked samples shall be prepared by adding a volume of a standard to a known volume of sample. To ensure adequate method detection limits, the volume of the standard added to the sample should be minimized to 5% or less of the volume of the sample. The spiked sample should be carried through the same sample preparation steps as the background sample.

(2) The percent recovery of spiked samples should be calculated as follows:

$$\%Recovery = \frac{100\ (C_c(V_o + V_s) - C_o V_o)}{C_s V_s}$$

where:

C_c=concentration of spiked sample
C_o=concentration of sample without spiking
C_s=known concentration of spiking standard

V_o=volume of sample
V_s=volume of spiking standard added to the sample

(3) If the percent recovery of a spiked sample is outside the range of 100% ±13% of the theoretical concentration for methanol and ethanol, or 100% ±16% for t-butanol and MTBE, the sources of error in the analysis should be determined, corrected, and all analyses subsequent to and including the last analysis confirmed to be within

the compliance specifications must be repeated. The specification limits for the accuracy of the percent recovery of spiked sample analyses are minimum performance requirements. The performance of individual laboratories may indeed be better than these minimum requirements. For this reason it is recommended that control charts be utilized to monitor the variability of measurements in order to optimally detect abnormal situations and ensure a stable measurement process.

(9)(i) Prior to January 1, 1997, and when the oxygenates present are limited to MTBE, ETBE, TAME, DIPE, tertiary-amyl alcohol, and C_1 to C_4 alcohols, any refiner, importer, or oxygenate blender may determine oxygen and oxygenate content using ASTM standard method D–4815–93, entitled "Standard Test Method for Determination of MTBE, ETBE, TAME, DIPE, tertiary-Amyl Alcohol and C_1 to C_4 Alcohols in Gasoline by Gas Chromatography," for purposes of meeting any testing requirement; provided that

(ii) The refiner or importer test result is correlated with the method set forth in paragraphs (g)(1) through (g)(8) of this section.

(h) *Incorporations by reference.* ASTM standard methods D–3606–92, D–1319–93, D–4815–93, D–2622–92, and D–86–90 with the exception of the degrees Fahrenheit figures in Table 9 of D–86–90, are incorporated by reference. These incorporations by reference were approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(A) and 1 CFR part 51. Copies may be obtained from the American Society of Testing Materials, 1916 Race Street, Philadelphia, PA 19103. Copies may be inspected at the Air Docket Section (LE–131), room M–1500, U.S. Environmental Protection Agency, Docket No. A–92–12, 401 M Street SW., Washington, DC 20460 or at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC.

§ 80.47 [Reserved]

§ 80.48 Augmentation of the complex emission model by vehicle testing.

(a) The provisions of this section apply only if a fuel claims emission reduction benefits from fuel parameters that are not included in the complex emission model or complex emission model database, or if the values of fuel parameters included in the complex emission model set forth in § 80.45 fall outside the range of values for which the complex emission model is deemed valid.

(b) To augment the complex emission model described at § 80.45, the following requirements apply:

(1) The petitioner must obtain prior approval from the Administrator for the design of the test program before beginning the vehicle testing process. To obtain approval, the petitioner must at minimum provide the following information: the fuel parameter to be evaluated for emission effects; the number and description of vehicles to be used in the test fleet, including model year, model name, vehicle identification number (VIN), mileage, emission performance (exhaust THC emission level), technology type, and manufacturer; a description of the methods used to procure and prepare the vehicles; the properties of the fuels to be used in the testing program (as specified at § 80.49); the pollutants and emission categories intended to be evaluated; the precautions used to ensure that the effects of the parameter in question are independent of the effects of other parameters already included in the model; a description of the quality assurance procedures to be used during the test program; the statistical analysis techniques to be used in analyzing the test data, and the identity and location of the organization performing the testing.

(2) Exhaust emissions shall be measured per the requirements of this section and § 80.49 through § 80.62.

(3) The nonexhaust emission model (including evaporative, running loss, and refueling VOC and toxics emissions) shall not be augmented by vehicle testing.

(4) The Agency reserves the right to observe and monitor any testing that is performed pursuant to the requirements of this section.

(5) The Agency reserves the right to evaluate the quality and suitability of data submitted pursuant to the requirements of this section and to reject, re-analyze, or otherwise evaluate such data as is technically warranted.

(6) Upon a showing satisfactory to the Administrator, the Administrator may approve a petition to waive the requirements of this section and § 80.49, § 80.50(a), § 80.60(d)(3), and § 80.60(d)(4) in order to better optimize the test program to the needs of the particular fuel parameter. Any such waiver petition should provide information justifying the requested waiver, including an acceptable rationale and supporting data. Petitioners must obtain approval from the Administrator prior to conducting testing for which the requirements in question are waived. The Administrator may waive the noted requirements in

whole or in part, and may impose appropriate conditions on any such waiver.

(c) In the case of petitions to augment the complex model defined at § 80.45 with a new parameter, the effect of the parameter being tested shall be determined separately, for each pollutant and for each emitter class category. If the parameter is not included in the complex model but is represented in whole or in part by one or more parameters included in the model, the petitioner shall be required to demonstrate the emission effects of the parameter in question independent of the effects of the already-included parameters. The petitioner shall also have to demonstrate the effects of the already-included parameters independent of the effects of the parameter in question. The emission performance of each vehicle on the fuels specified at § 80.49, as measured through vehicle testing in accordance with § 80.50 through § 80.62, shall be analyzed to determine the effects of the fuel parameter being tested on emissions according to the following procedure:

(1) The analysis shall fit a regression model to the natural logarithm of emissions measured from addition fuels 1, 2, and 3 only (as specified at § 80.49(a)) and adjusted as per paragraph (c)(1)(iv) of this section that includes the following terms:

(i) A term for each vehicle that shall reflect the effect of the vehicle on emissions independent of fuel compositions. These terms shall be of the form $D_i \times V_i$, where D_i is the coefficient for the term and V_i is a dummy variable which shall have the value 1.0 for the ith vehicle and the value 0 for all other vehicles.

(ii) A linear term in the parameter being tested for each emitter class, of the form $A_i \times (P_1 - P_1 (avg)) \times E_i$, where A_i is the coefficient for the term, P_1 is the level of the parameter in question, P_1 (avg) is the average level of the parameter in question for all seven test fuels specified at § 80.49(a)(1), and E_i is a dummy variable representing emitter class, as defined at § 80.62. For normal emitters, $E_1=1$ and $E_2=0$. For higher emitters, $E_1=0$ and $E_2=1$.

(iii) For the VOC and NO_x models, a squared term in the parameter being tested for each emitter class, of the form $B_i \times (P_1 - P_1 (avg))^2 \times E_i$, where B_i is the coefficient for the term and where P_1, P_1 (avg), and E_i are as defined in paragraph (c)(1)(ii) of this section.

(iv) To the extent that the properties of fuels 1, 2, and 3 which are incorporated in the complex model differ in value among the three fuels, the